Not for Publication
Presented Before the Division of Gas and Fuel Chemistry
American Chemical Society
Boston, Massachusetts, Meeting, April 5-10, 1959

The Relation of the Chemical and Physical Properties of Coal Tar Pitches to their Carbonization and Graphitization Character

P.L. Walker, Jr., C.R. Kinney, D.O. Baumbach, and M.P. Thomas
Fuel Technology Department, The Pennsylvania State University
University Park, Pennsylvania

INTRODUCTION

Coal tar pitch, derived from by-product ovens, is the preferred material for use as a binder in the manufacture of carbon and graphite electrodes. Variations in the type of coke oven and coke oven practice and the diversity of sources of supply give rise to variations in pitch quality which affect electrode performance and result, in the case of some pitch binders, in poor performance. At the moment, it seems in vogue for industry to invariably blame poor electrode performance on the binder, despite variations in the properties of the filler (usually petroleum coke), which may be as great as those variations in binder properties. It appears that the main justification for attributing poor electrode performance to the binder is the feeling that coal tar pitch coke does not grow in crystallite size as markedly as does petroleum coke upon heat treatment. With the attendant smaller growth in crystallite size, it is suggested that the pitch binder possesses inferior quality for such important properties as electrical and thermal conductivity, reactivity to gases, coefficient of thermal expansion, and strength.

The three initial aims of this research have been: (1) to seek relationships between the chemical and physical properties of coal tar pitches and the properties of the cokes and graphitized carbons produced therefrom; (2) to better understand the effect of heat treatment temperature on the properties of carbons produced from coal tar pitch; and (3) to compare the properties of the cokes and graphitized carbons produced from coal tar pitch with those produced from a standard, commercially used petroleum coke. For reaction rate studies, comparisons are also made with a coal coke and lampblack. This paper presents the results on this initial phase of research.

Work is now in progress studying the interaction between the filler and binder phases. Selected pitches from those examined in the initial phase of the research are being processed with a standard petroleum coke in a conventional manner to produce carbon and graphite bodies. Important properties of the bodies will be compared with the same properties of the cokes and graphites produced separately from the pitches in an attempt to learn the real significance of binder properties in affecting electrode performance. The results of this latter phase of research will be reviewed at a future date.

EXPERIMENTAL

A. Preparation of Coke and Graphitized Carbons

1. Charring of the coal tar pitches. A sample of 250 grams of coal tar pitch was placed in an annealed glass liner (5.4 cm. o.d. and 52.5 cm. long). The liner was enclosed in a gas-tight steel bomb (1780 cc. capacity) and the bomb was evacuated prior to filling with nitrogen to atmospheric pressure. The bomb was heated to 550±2.5°C. in twelve hours and held at 550°C. for an additional twelve hours. The maximum pressure developed on charring ranged from 710 to 890 psig., depending upon the pitch. The bomb was allowed to cool to room temperature before opening.

The recovery of char inside the glass liner, which was subsequently coked and graphitized, generally ranged from 67 to 78 per cent of the initial pitch weight. The remainder of the weight could be accounted for by char between the glass liner and bomb wall and by permanent gases. The molecular weight of the permanent gases was estimated in several runs to be about 22. Two charring runs were made on each pitch, with the charring yields agreeing consistently within 1.5 per cent.

2. Grinding of the coal tar pitch chars. The pitch-chars were ground in a hammer mill and screened through U.S. sieves 40 and 60 mesh. The 40 x 60 mesh fraction, which amounted to between 30 and 40 per cent of the char yield, was used in the subsequent coking operation.

3. Coking of the coal tar pitch chars. Twenty-five grams of char from each charring run were heated (in a Vycor tube flushed with helium) at a rate of 5°C. per minute to 1000°C. and soaked for one hour at this temperature. Variation in temperature along the sample length was less than ±6°C. The coke yield amounted to about 65 to 70 per cent of the initial pitch weight. Negligible fusing of the 40 x 60 mesh char particles was found on coking.

4. Graphitization of the coal tar pitch cokes. Approximately 10 grams of 40 x 60 mesh coke were placed in cylindrical graphite capsules (1 1/2 in. o.d. and 1 1/2 in. long). The capsules were loaded into a larger cylindrical graphite capsule (4 in. o.d. by 11 in. long), which was then centered in a resistance furnace. The resistance furnace, which was flushed with helium during a run, consisted of a graphite tube 6 inches in diameter and several feet long. Samples were allowed to cool to room temperature before being removed. All temperatures were measured with a Pyro optical pyrometer. Samples graphitized by resistance heating are designated as carbons (R).

5. Additional heat treatment of a variety of carbons. In order to understand the effects of a wide heat treatment range on the subsequent reactivity of coal tar pitch coke, petroleum coke, coal coke, and lampblack to oxidizing gases, samples were heated to temperatures of approximately 1750, 2000, 2250, 2600, and 2800°C. in an induction furnace, with zero soak time at the maximum temperature. Three cylindrical graphite crucibles (7/8 in. o.d. and 1 1/2 in. long) were partially filled with 5 grams of sample. The crucibles were inserted into a graphite tube (1 1/4 in. o.d. and 21 in. long), which in turn was centered into a quartz tube (3 1/4 in. o.d. and 25 1/4 in. long). The furnace was evacuated over night at room temperature and then flushed with helium during the run. The temperature was regulated by manual control of a transformer. Temperature readings were taken with an optical pyrometer sighted through a quartz window at the top of the furnace.

B. Apparatus and Procedures Used to Define the Properties of the Pitches, Cokes, and Graphitized Carbons

Standard procedures previously described were used in the x-ray diffraction (2), surface area (3,4), electrical resistivity (5), and gas reactivity studies (6). Analyses of the coal tar pitches were determined using standard specification studies (7).

C. Description of Raw Materials

The coal tar pitches represent the residual product from the distillation of high-temperature coke-oven tars. No information is available on the carbonization temperatures at which the tars were produced or the distillation procedures used to produce the coal tar pitches.

Raw petroleum coke is the coked still bottoms left in the pot after distillation of the crude oil. The raw coke having seen a temperature of ca. 500°C. was calcined at ca. 1250°C. It had an ash content of 0.12 per cent and a carbon content of 97.8 per cent.

Lampblack is prepared from the partial combustion of liquid hydrocarbons. The lampblack used in this research was taken from crushed, 5/8-in. gas baked (ca. 1000°C.) lampblack electrodes. The green electrodes contained a mixture of 75 per cent lampblack and 25 per cent coal tar pitch by weight. The baked material had an ash content of 0.22 per cent and a carbon content of 98.9 per cent.

The coal coke was a standard low temperature foundry coke produced at about 600°C. It had an ash content of 6.8 per cent and a carbon content of 85.0 per cent.

RESULTS AND DISCUSSION

A. Analyses of the Coal Tar Pitches

Table I presents data on the chemical analyses of the whole pitches. The analysis for sample BD-PSU-5 is markedly different from the remaining samples, since it is a lignite pitch.

Table II presents semi-quantitative data on the concentration of metallic impurities in some selected pitches. The concentrations were determined using standard spectrochemical techniques. The concentrations are expressed in parts of impurity per million parts of pitch on a weight basis.

Table III presents miscellaneous data on the pitches. The coking values were determined using the Barrett Method B-8 (7), which consists of rapidly heating the pitch to 900°C. and holding the temperature for seven minutes. The softening point of the pitches was determined by the ring and ball method (7). From Table III, it is seen that the specific gravity of the whole pitch increases with an increase in its quinoline insoluble content. Also as expected, it is seen from Table III that as the specific gravity increases, the coking value also increases.

B. Chemical Analyses of the Cokes from the Group A Coal Tar Pitches

Table IV presents data on the chemical analyses of the cokes from the Group A* coal tar pitches. For these pitches, two charring runs and three coking runs were made, with the char from one run being coked twice. The data, consequently, represent an average of three coking runs. The amount of hydrogen in the original pitch removed upon coking varies from 81 per cent for sample 22805 to 90 per cent for sample 23217. The amount of nitrogen removed from the pitch ranges from 28 per cent for sample 23217 to 100 per cent for sample 56-273. The amount of sulfur removed from the pitch ranges from 28 per cent for sample 22761 to 54 per cent for sample 56-274. Thus, there is considerable variation in the amount of hydrogen, nitrogen, and sulfur removed on coking of the different pitches. However, there is no obvious correlation between the amount of these elements in the original pitch and the percentage of them removed on coking.

C. Crystallographic Parameters for the Chars, Cokes, and Graphitized Carbons from the Group A Coal Tar Pitches

1. Interlayer spacings and crystallite heights. Table V presents data on the interlayer spacings and crystallite heights of the chars, cokes, and graphitized carbons produced from the Group A coal tar pitches. X-Ray diffraction determinations were made on a sample from each run. Consequently, the data on the chars represent the average of two determinations; on the cokes, an average of three determinations; and on the graphitized carbons, an average of two determinations at each temperature (that is, two different samples of coke were heated in each graphitization run). The graphitization runs were performed in the resistance furnace previously described. The interlayer spacing data in all cases are significant to ±0.02A., ±0.01A., and ±.001A. for the chars, cokes, and graphitized carbons, respectively. The crystallite height data in all cases are significant to ±1A., ±1A., and ±50A., respectively.

Franklin (8) and Bacon (9) have thoroughly discussed the use of interlayer spacing data as a criterion of three-dimensional ordering in carbon. Interlayer spacings above 3.44A. are indicative of only two dimensional ordering, whereas decreasing spacings between 3.44A. and 3.3538A. indicate a progressive increase in three-dimensional ordering between zero and 100 per cent. As expected, the interlayer spacing data on the chars and cokes indicate no three-dimensional ordering. Furthermore, for each group the spacings are essentially the same. In line with the findings of Kinney, Nunn, and Walker (10), among others, the interlayer spacings of the material having seen 1000°C. (cokes) are somewhat larger than the comparable material having seen only 550°C. (chars). As expected, the calcined petroleum coke having seen a temperature of ca. 1250°C. has a lower interlayer spacing than the cokes from the coal tar pitches. That is, the interlayer spacing of carbons begins to decrease markedly at heat treatment temperatures above about 1050°C.

* The first six coal tar pitches, on which some additional work was done, will be called Group A pitches hereafter. The remainder of the pitches will be called Group B.

On graphitization, a marked decrease in interlayer spacing of all the cokes is seen. The effect of increasing graphitization temperature on further decreasing the interlayer spacing is also evident, as previously discussed (11). The carbon produced from pitch 23217 by graphitization at 2650°C. is seen to have the lowest interlayer spacing, 3.359A., and, consequently, the highest degree of three-dimensional ordering, ca. 85 per cent. The petroleum coke graphitized at 2570°C. has a lower interlayer spacing than the majority of the pitch cokes graphitized at the same temperature. Unfortunately, the petroleum coke was not heat treated at 2650°C.

From Table V, it is seen that there is little significant difference in the average crystallite heights between the chars, as a group or between the cokes, as a group. Some decrease in the average crystallite sizes of the cokes over the comparable chars are noted, in line with previous findings (10). The petroleum coke has a considerably higher crystallite height than any of the pitch cokes, as expected because of its higher calcination temperature. Extensive increases in average crystallite heights of the pitch cokes on graphitization are noted. However, the effect of increasing the graphitization temperature from 2570 to 2650°C. on increasing the crystallite heights is not indicated clearly by the data. In any event, the marked decrease in interlayer spacing and increase in crystallite height upon heating to graphitization temperatures clearly stamp these pitch cokes as "graphitizable carbons".

2. Orientation of crystallites in pitch cokes. As pointed out, the interlayer spacing and crystallite height data on the pitch cokes show such minor variations that it is difficult to distinquish between the samples. A similar situation was shown to exist when a series of calcined petroleum cokes was examined by x-ray diffraction (12). However, in the latter case it was shown that the extent of crystallite orientation in the cokes, as given by the intensity of the (002) x-ray diffraction peak, varied considerably and could be used to predict important properties of carbon bodies made from the cokes. Consequently, relative intensities of the (002) peak for the pitch cokes were determined using techniques previously described (2,12). Peak intensities were determined on the three coke samples from each pitch, and one additional intensity determined on a second sample of one of the cokes selected at random. The average peak intensity for the cokes from each pitch varied in all cases by less than ±10 per cent. The intensities varied from 24.4 c.p.s. for pitch coke 22761 (poorest crystallite orientation) to 34.4 c.p.s. for pitch coke 23217 (best crystallite orientation).

D. Surface Areas of the Chars, Cokes, and Graphitized Carbons from the Group A Coal Tar Pitches

Table VI presents surface area data on 40 x 60 mesh samples of coke and graphitized carbons produced from the Group A coal tar pitches. The surface area for the char produced from pitch 23217 is also included. Determinations were made on the same number of samples as in the x-ray diffraction studies, with the average surface areas reported in Table VI representing all samples from a given pitch within at least ±10 per cent.

The first major conclusion to be drawn from the data in Table VI is that the surface areas of the cokes (and char) are quite small. These small areas indicate that the cokes (and char) contain a negligible amount of open porosity in fine pores. A small area is desirable for a graphitizable carbon since it means that there will be a minimum of discontinuities within the particle to stop crystallite growth. It should be noted that the petroleum coke has a somewhat higher area than the coal tar pitch cokes. This could be due entirely, or in part, to the higher heat treatment temperature of the petroleum coke, since it has been shown that surface areas of some carbons do increase slightly when heated in the range 950 to 1100°C.

No relationship is found between the surface area of the cokes and their graphitizability. For example, carbon from pitch 23217 has the lowest interlayer spacing on heat treatment to 2650°C. but yet does not have the lowest surface area, as seen in Table VI. This lack of correlation is not surprising if the very low values of the surface areas are kept in mind. At 2650°C., the extent of crystallite growth is probably not yet limited by the infrequent pores in the coke particle (11).

From Table VI, it is seen that there is a decrease in the specific surface areas of the cokes on heat treatment to 2570 and 2650°C. At 2650°C., this decrease ranges from 50 per cent for sample 22761 to 13 per cent for sample 22763. The percentage decrease in surface area is seen to be roughly related to the surface area of the coke - the higher its area the greater the percentage decrease. This results in the surface areas of all the graphitized carbons varying much less than the surface areas of the cokes.

Despite the over-all decrease in areas on graphitization, there is some indication that the specific surface area of the graphitized carbons increase slightly in going from 2570 to 2650°C. Perhaps volatilization of some impurities in this temperature range produced slight porosity. It is noted that the surface area of the petroleum coke after graphitization at 2570°C. is ca. 25 per cent greater than the surface area of the graphitized pitch cokes.

Looking at the chemical analyses of the coal tar pitches in Table I, no correlation is found between the percentage of hydrogen, nitrogen, or sulfur in the pitch and the surface areas of the cokes or graphitized carbons produced therefrom.

E. <u>Electrical Resistivities of Cokes and Graphitized Carbons</u>

Table VII presents electrical resistivity data on the cokes and graphitized carbons produced from both the Group A and B coal tar pitches. The resistivities of the cokes are seen to be quite similar. Disregarding samples 22761 and BD-PSU-5 (lignite pitch), the resistivities of the remaining pitch cokes vary by only ±12 per cent. No correlation is found between the electrical resistivities of the Group A cokes and their crystallographic parameters or surface areas. Presumably, likewise no correlations would exist for the Group B pitch cokes. As expected because of its higher calcination temperature, the petroleum coke has a lower electrical resistivity than any of the pitch cokes.

On graphitization, there is a marked decrease in the electrical resistivities of all samples. Further, as expected (13), the higher temperature graphitization run* produced, for each sample, the lower electrical resistivity. As for the cokes, no correlation exists between the electrical resistivities of the graphitized carbons from the Group A pitches and the crystallographic parameters or surface areas of the carbons.

The electrical resistivities for the graphitized petroleum coke are comparable with those for many of the graphitized pitch cokes heated to the same temperature. Actually, the electrical resistivities of the graphitized pitch cokes from samples 23217, 56-274, BD-PSU-8, -12, -16, and -17 are lower than the resistivities of the graphitized petroleum coke.

F. Gas Reactivities of Cokes and Graphitized Carbons

1. Reactivities of cokes and graphitized carbons to carbon dioxide at 1150°C. Tables VIII and IX present data for the reactivity of cokes and graphitized carbons from the Group A and Group B pitches, respectively. Clearly, for the majority of the pitch samples, the reactivity of the carbons graphitized between 2570 and 2660°C. is greater than the reactivity of the cokes. Samples 56-274 (graphitized at 2570°C.) and BD-PSU-2 are exceptions. For the Group A coal tar pitches, an increase in graphitization temperature from 2570 to 2650°C. results in a marked increase in reactivity of the carbons to carbon dioxide. To the contrary, for the Group B coal tar pitches (and pitch 22805 from Group A) an increase in graphitization temperature from 2660 to 2680°C. produces a decrease in reactivity, with two exceptions. These exceptions are samples BD-PSU-1 and -18.

It is noted that the wide variation in reactivities of the pitch cokes to carbon dioxide is not carried over to the graphitized samples. For example, considering the Group B pitches, the cokes show a spread in reactivity from 3.5 to 30.8 per cent burn-off in two hours. On the other hand, exclusive of BD-PSU-5 (lignite pitch), the samples graphitized at 2660°C. show a spread in reactivity from only 19.8 to 28.1 per cent.

A qualitative correlation is found between the per cent ash in the coal tar pitches (Table I) and the reactivity of the cokes produced therefrom (Table VIII and IX). Even though there is considerable scatter in the data, in general, higher ash content in the pitch means higher reactivity of the coke towards carbon dioxide. The scatter in the data is to be expected since individual constituents of the impurity phase will have different effects on increasing (or decreasing) the reactivity of the coke. No correlation is found, however, between the concentrations of any individual constituent of the impurity phase in the pitch and reactivity of the cokes.

For the Group A coal tar pitches, the reactivity data of the cokes (as a group) or graphitized carbons (as a group) cannot be explained on the basis of the surface area or crystallographic parameters of these materials.

---

* It is noted that the majority of the Group A samples were graphitized at temperatures lower than those used for the Group B samples. Unfortunately, insufficient samples of the Group A cokes were available to make heat treatment runs at 2660 and 2680°C.

In marked contrast to the increase in reactivity upon graphitization for the majority of the pitch samples, the petroleum coke is seen to undergo a major decrease in reactivity upon graphitization to 2570°C. Further, the reactivity of the petroleum coke is many-fold greater than the reactivity of the majority of the pitch cokes; whereas the reactivity of the graphitized petroleum coke is comparable to that of many of the graphitized pitch cokes.

2. <u>Reactivities of different carbons to carbon dioxide and air as a function of heat treatment temperature.</u> Samples of pitch coke 56-273, petroleum coke, lampblack, and coal coke were heat treated in a helium atmosphere at a series of temperatures, using an induction furnace. Separate runs also were made at selected temperatures on pitch coke 56-273 and petroleum coke in an induction furnace, using a 1 per cent chlorine-99 per cent helium atmosphere. Because of space limitations in the induction furnace, only sufficient sample could be heat treated to enable the making of one reactivity run, each, in carbon dioxide and air. Table X summarizes the data.

In agreement with previous findings where the resistance furnace was used to heat treat the samples, the reactivity of the pitch coke is seen to increase upon being treated to higher temperatures. A maximum in reactivity is found for heat treatment to ca. 2000°C. Another maximum (displaced to a higher temperature than found previously for sample 22805) probably occurs at a temperature above 2800°C. Also as found before, the reactivity of petroleum coke to carbon dioxide shows a marked decrease upon heat treatment. The initial decrease is found to occur between the calcination temperature and a heat treatment temperature of 1750°C. A slight increase in reactivity is suggested with an increase in heat treatment temperature between 1750 and 2250°C. This is followed by a second, sharp decrease in reactivity on heat treatment to 2600°C. No further maximum in reactivity is found for the petroleum coke between 2600 and 2800°C., probably because the gap in heat treatment temperature was too large to uncover a maximum.

Limited data indicate that the success attained in decreasing carbon reactivity by heat treatment in a partial chlorine atmosphere depends upon the carbon and heat treatment temperature. For example, for the pitch coke 56-273, it is seen that the use of a partial chlorine atmosphere during heat treatment at ca. 1750 and 2000°C. markedly decreases the subsequent reactivity of the carbon to carbon dioxide. On the other hand, the use of chlorine at ca. 2250°C. shows little effect. The use of a partial chlorine atmosphere during the heat treatment of petroleum coke at ca. 2000 and 2250°C. also shows little effect on the subsequent reactivity.

It is seen from Table X that heat treatment of lampblack and coal coke over a range of temperatures has a negligible effect on the subsequent reactivity of these materials to carbon dioxide. This is particularly surprising in the case of the coal coke, which undoubtedly underwent a major decrease in ash content on heat treatment at 2600°C.

The effect of heat treatment on the reactivity of pitch coke and petroleum coke to air is seen to be considerably different from the effect of heat treatment on their reactivity to carbon dioxide. The reactivity of both these materials to air is seen to decrease progressively with increasing heat treatment temperature. Furthermore, the reactivities of the two materials to air for heat treatment temperatures up to 2250°C. parallel each other closely. The use of a partial chlorine atmosphere is seen to decrease markedly the subsequent reactivity of the pitch coke to air at all heat treatment temperatures investigated.

As in the carbon-carbon dioxide reaction, variation in heat treatment temperature is seen to have a negligible effect on the reactivities of lampblack and coal coke to air.

It is readily apparent that the reactivity of carbons to carbon dioxide is much more sensitive to carbon-type than is the reactivity of carbons to air. In the latter case, it is seen that the reactivities of different carbons, following particular heat treatment temperatures, are quite similar.

ACKNOWLEDGEMENTS

The financial support of the Plastics and Coal Chemicals Division of the Allied Chemical Corporation made this work possible. Their support is gratefully acknowledged. Sincere appreciation is extended to Messrs. W.M. Bywater and J.F. Weiler of the above organization for following the entire research program closely, offering invaluable advice, and supplying much pertinent analytical data on the pitches. Special appreciation is extended to the Speer Carbon Company for graphitizing the cokes in their experimental resistance furnace.

REFERENCES

1. M.M. Stimson and M.J. O'Donnel, J. Am. Chem. Soc., 74, 1805 (1952).
2. P.L. Walker, Jr., H.A. McKinstry, and J.V. Pustinger, Ind. Eng. Chem., 46, 1651 (1954).
3. P.H. Emmett, A.S.T.M. Tech. Publ., 51, 95 (1941).
4. P.L. Walker, Jr. and C.C. Wright, Ind. Eng. Chem., 45, 1715 (1953).
5. P.L. Walker, Jr. and F. Rusinko, Jr., Fuel 36, 43 (1957).
6. P.L. Walker, Jr. and J.R. Nichols, Proceedings of the Industrial Carbon and Graphite Conference (London), pp. 334-347 (1958).
7. "Methods of Treating Coal-Tar Products", Plastics and Coal Chemicals Division, Allied Chemical Corp., 1950.
8. R.E. Franklin, Acta Cryst., 4, 253 (1951).
9. G.E. Bacon, Acta Cryst., 4, 558 (1951).
10. C. R. Kinney, R. C. Nunn, and P.L. Walker, Jr., Ind. Eng. Chem., 49, 880 (1957).
11. P.L. Walker, Jr. and G. Imperial, Nature, 180, 1184 (1957).
12. P.L. Walker, Jr., F. Rusinko, Jr., J.F. Rakszawski, and L.M. Liggett, "Effect of Different Cokes on Physical Properties of Graphitized Carbon Plates", Presented at the Third Conference on Carbons, Univ. of Buffalo, June 17-21, 1957, in press.
13. H.T. Pinnick, Proceedings of the First and Second Conferences on Carbon, Buffalo, N.Y., 1956, pp. 3-12.

TABLE I

CHEMICAL ANALYSES OF THE PITCHES

Pitch	%C	%H	%N	%S	%Other*	%Ash	Atomic C-H Ratio
22761	93.32	4.20	0.75	0.75	0.98	0.10	1.86
22763	93.43	4.24	0.68	0.52	1.13	0.09	1.85
22805	92.43	4.31	0.88	0.41	1.97	0.24	1.80
23217	92.05	4.52	0.50	0.54	2.39	0.13	1.71
56-273	93.39	4.31	0.92	0.60	0.78	0.08	1.82
56-274	92.95	4.49	0.86	0.35	1.35	0.15	1.74
BD-PSU-1	93.35	4.32	1.08	0.27	0.98	0.17	1.81
-2	91.70	4.55	1.31	0.53	1.91	0.63	1.69
-3	92.77	4.32	1.21	0.58	1.12	0.07	1.80
-4	93.34	4.10	1.10	0.49	0.97	0.05	1.91
-5	81.67	7.31	1.16	0.77	9.09	2.55	0.94
-6	93.34	4.38	1.01	0.30	0.97	0.12	1.79
-7	93.36	4.44	1.00	0.31	0.89	0.14	1.77
-8	93.26	4.40	1.01	0.24	1.09	0.11	1.78
-9	93.18	4.46	1.00	0.32	1.04	0.10	1.76
-10	93.47	4.44	0.98	0.25	0.86	0.05	1.77
-11	93.25	4.49	0.99	0.31	0.96	0.05	1.74
-12	93.31	4.46	1.01	0.31	0.91	0.04	1.76
-13	93.17	4.57	1.00	0.30	0.96	0.03	1.71
-14	93.00	4.20	1.06	0.41	1.33	0.25	1.86
-15	93.08	4.29	1.06	0.47	1.10	0.22	1.82
-16	92.24	4.53	1.11	0.39	1.03	0.06	1.72
-17	92.78	4.65	1.10	0.37	1.10	0.06	1.68
-18	93.13	4.42	1.08	0.37	1.00	0.08	1.77
-19	93.13	4.49	1.11	0.37	0.90	0.08	1.74

* Includes % Ash

TABLE II

CONCENTRATION OF METALLIC IMPURITIES IN SELECTED PITCHES

CONCENTRATION, PPM

Pitch	Al	B	Ca	Cu	Fe	Mg	Mn	Na	Ni	Pb	Si	Ti
22761	7.4	-	8.4	2.0	128.	2.2	2.8	98.	-	23.	41.	-
22763	6.9	-	3.5	4.5	44.	2.2	1.6	40.	-	22.	17.	-
22805	72.	-	99.	19.	98.	9.6	4.3	206.	-	53	96.	-
23217	22.	-	9.6	1.8	46.	5.2	2.3	220.	-	23.	43.	-
56-273	17.	-	25.	1.3	35.	4.2	2.5	31.	-	19.	72.	-
56-274	36.	-	22.	7.3	116.	12.	9.3	78.	-	69.	48.	-
BD-PSU-2	310.	0.88	110.	5.0	360.	23.	11.	-	21.	11.	510.	25.
-4	12.	0.14	6.4	1.3	58.	2.0	1.2	-	2.8	8.5	120.	1.4
-5	2400.	67.	160.	15.	1200.	180.	160.	-	33.	72.	3100.	260.
-6	100.	0.14	46.	1.6	96.	6.4	7.4	46.	4.5	34.	130.	7.4
-9	97.	0.085	23.	0.92	79.	6.7	4.4	28.	3.0	58.	120.	9.4
-11	28.	0.019	13.	0.63	43.	1.6	1.7	37.	1.8	26.	27.	2.3
-12	27.	0.020	17.	0.90	40.	2.8	4.6	42.	3.5	35.	60.	6.0
-13	59.	0.031	52.	1.1	90.	3.9	9.8	90.	6.1	59.	57.	6.2
-14	170.	0.12	75.	2.9	180.	16.	13.	57.	4.2	68.	360.	16.
-16	24.	0.0062	23.	0.48	86.	3.4	5.8	39.	1.8	56.	22.	1.0
-18	28.	0.016	13.	0.62	98.	3.4	5.2	32.	2.0	41.	43.	1.9
-19	62.	0.028	120.	0.80	150.	7.0	8.5	50.	2.9	60.	100.	4.3

TABLE III

MISCELLANEOUS PROPERTIES OF THE PITCHES

Pitch	Coking Value	Sp. Gr., 25°C.	Soft. Pt., °C.	% Insoluble in Quinoline	% Insoluble in Benzene
22761	44.5	1.328	95.5	14.9	32.1
22763	43.8	1.333	98.0	16.5	36.4
22805	41.0	1.315	95.0	12.8	27.1
23217	44.2	1.292	96.0	7.8	25.2
56-273	43.0	1.320	95.0	13.9	33.2
56-274	39.2	1.305	99.9	12.5	32.0
BD-PSU-1	42.8	1.318	94.6	15.8	32.5
-2	35.7	1.298	100.0	5.7	25.5
-3	40.0	1.318	96.0	13.3	27.1
-4	41.7	1.340	101.8	13.4	27.8
-5	24.8	1.174	103.0	8.6	18.9
-6	41.5	1.313	95.2	13.0	29.4
-7	39.8	1.306	88.7	12.2	28.4
-8	37.5	1.307	94.5	10.2	22.5
-9	36.2	1.300	89.5	9.9	20.2
-10	40.5	1.308	94.7	11.6	28.3
-11	37.1	1.297	87.0	8.8	23.7
-12	36.1	1.299	95.0	7.6	19.6
-13	33.3	1.292	88.5	6.0	17.9
-14	42.3	1.328	96.5	18.1	28.6
-15	40.2	1.314	88.2	17.2	27.2
-16	34.7	1.294	97.0	5.2	17.5
-17	32.3	1.286	85.5	5.1	16.3
-18	40.1	1.306	99.0	10.4	26.9
-19	36.1	1.296	87.5	8.3	24.2

TABLE IV

CHEMICAL ANALYSES OF THE COKES FROM THE GROUP A COAL TAR PITCHES

Coke	%C	%H	%N	%S	% Other	Atomic C-H Ratio
22761	98.16	0.48	0.23	0.54	0.57	17.2
22763	97.68	0.61	0.03	0.35	0.35	13.4
22805	96.53	0.79	0.16	0.26	0.26	10.6
23217	97.96	0.44	0.36	0.26	0.26	18.5
56-273	98.35	0.56	0.00	0.29	0.29	14.1
56-274	98.11	0.75	0.05	0.16	0.16	11.4

TABLE VI

SURFACE AREAS OF THE CHARS, COKES, AND GRAPHITIZED CARBONS PRODUCED FROM THE GROUP A COAL TAR PTICHES

Pitch	Surface Area, m^2/g			
	Char	Coke	Graphitized Carbon	
			2570°C.	2650°C.
22761	-	0.36	0.18	0.18
22763	-	0.24	0.17	0.21
22805	-	0.26	0.17	0.20
23217	0.38	0.34	0.23	0.21
56-273	-	0.31	0.19	0.20
56-274	-	0.45	0.24	0.28
petroleum coke	-	0.59	0.34	-

TABLE V

INTERLAYER SPACINGS AND CRYSTALLITE HEIGHTS OF THE CHARS, COKES, AND GRAPHITIZED CARBONS PRODUCED FROM THE GROUP A COAL TAR PITCHES

	Interlayer Spacing, A.				Crystallite Height, A.			
	Char	Coke	Graphitized Carbon		Char	Coke	Graphitized Carbon	
Pitch			2570°C.	2650°C.			2570°C.	2650°C.
22761	3.54	3.58	3.370	3.363	18	14	618	613
22763	3.56	3.57	3.367	3.362	16	15	686	710
22805	3.55	3.57	3.369	3.364	17	14	653	702
23217	3.52	3.55	3.365	3.359	19	15	734	895
56-273	3.53	3.56	3.367	3.363	17	15	(789)	720
56-274	3.53	3.56	3.365	3.362	19	14	738	904
petroleum coke	-	3.51	3.364	-	-	25	-	-

TABLE VII

ELECTRICAL RESISTIVITIES AT 2500 PSI. AND 25°C.
FOR COKES AND GRAPHITIZED CARBONS

Sample	Electrical Resistivity, ohm-cm.		
	Coke	Graphitized Carbon	
		2660°C.	2680°C.
22761	0.050	0.0081[a]	-
22763	0.038	0.0049[a]	-
22805	0.040	0.0042	0.0032
23217	0.038	0.0029[b]	-
56-273	0.038	0.0044[a]	-
56-274	0.032	0.0029[b]	-
BD-PSU-1	0.037	0.0032	0.0031
-2	0.035	0.0035	0.0034
-3	0.041	0.0046	0.0038
-4	0.038	0.0042	0.0032
-5	0.049	0.0050	0.0042
-6	0.036	0.0032	0.0030
-7	0.040	0.0030	0.0029
-8	0.038	0.0028	0.0025
-9	0.039	0.0037	0.0029
-10	0.037	0.0036	0.0027
-11	0.035	0.0031	0.0024
-12	0.037	0.0027	0.0026
-13	0.037	0.0034	0.0027
-14	0.040	0.0038	0.0031
-15	0.041	0.0039	0.0030
-16	0.034	0.0026	0.0023
-17	0.034	0.0020	0.0021
-18	0.032	0.0033	0.0023
-19	0.036	0.0035	0.0029
petroleum coke	0.024	0.0031	0.0025

[a] Graphitized at 2570°C. [b] Graphitized at 2650°C.

TABLE VIII

REACTIVITIES OF COKES AND GRAPHITIZED CARBONS FROM GROUP A PITCHES TO CARBON DIOXIDE AT 1150°C.

Sample	Weight Per Cent Burn-Off in Two Hours		
	Coke	Graphitized Carbon	
		2570°C.	2650°C.
22761	8.7[a]	16.2[b]	29.3
22763	9.8	12.4	24.4
22805	8.8	15.0	26.2
23217	7.4	17.6	24.3
56-273	7.6	12.4	31.1
56-274	22.0	15.9	27.0
petroleum coke	55.2	10.7	-

[a] Reactivities on pitch cokes represent average of reactivities determined on individual samples from three different coking runs.

[b] Reactivities on carbons at each graphitization temperature represent average of reactivities on two graphitized coke samples.

TABLE IX

REACTIVITIES OF COKES AND GRAPHITIZED CARBONS FROM THE GROUP B PITCHES TO CARBON DIOXIDE AT 1150°C.

Sample	Weight Per Cent Burn-Off in Two Hours: Coke	Graphitized Carbon: 2660°C.	Graphitized Carbon: 2680°C.
BD-PSU-1	17.7[a]	24.5[b]	26.8
-2	30.8	21.1	20.5
-3	8.7	23.0	19.1
-4	5.0	22.9	12.3
-5	30.2	40.3	37.9
-6	16.8	20.1	14.4
-7	15.0	20.5	14.7
-8	11.3	24.3	16.8
-9	14.0	25.0	21.9
-10	8.1	19.8	14.2
-11	8.7	23.8	17.3
-12	6.6	23.6	15.1
-13	3.5	21.7	14.5
-14	16.7	28.1	17.2
-15	17.5	25.7	17.9
-16	14.4	25.1	15.2
-17	11.6	20.8	15.2
-18	12.4	20.0	25.5
-19	14.1	22.7	16.8
22805	8.8	26.8	17.2
petroleum coke	55.2	22.5	15.2

[a] Reactivities on pitch cokes represent average of reactivities determined on individual samples from two different coking runs.

[b] Represents one reactivity run on one graphitized coke sample at each temperature in every case.

TABLE X

REACTIVITIES OF CARBONS TO CARBON DIOXIDE AND AIR

Sample	Heat Treatment Temperature, °C.	Weight Per Cent Burn-Off in Two Hours: Carbon Dioxide at 1150°C.	Air at 700°C.
Pitch 56-273	1000	7.6	25.8
"	1745	27.0	22.8
"	1750[a]	18.5	20.4
"	2000	28.3	21.3
"	2005[a]	12.3	12.4
"	2240	11.9	16.1
"	2250[a]	10.9	10.4
"	2600	6.4	10.4
"	2800	24.5	8.2
Petroleum Coke	1250	55.2	24.8
"	1750	34.4	22.5
"	2020	34.9	20.6
"	2005[a]	34.6	-
"	2250	37.0	16.2
"	2265[a]	34.6	-
"	2600	10.6	-
"	2825	11.0	14.4
Lampblack	1000	56.1	26.4
"	2270	57.7	25.5
"	2600	52.3	28.3
Coal Coke	600	49.2	26.5
"	2250	60.5	24.4
"	2600	55.6	25.8

[a] Atmosphere in furnace 1% Cl_2-99% He